International Standard 3634

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Vegetable products — Determination of chloride content

Produits dérivés des légumes — Détermination de la teneur en chlorures

First edition — 1979-07-15

UDC 635 : 543.848

Ref. No. ISO 3634-1979 (E)

Descriptors : vegetable products, chemical analysis, chlorides, determination of content.

Price based on 3 pages

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3634 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in May 1978.

It has been approved by the member bodies of the following countries :

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No member body expressed disapproval of the document.

Printed in Switzerland

INTERNATIONAL STANDARD **ISO 3634-1979 (E)**

Vegetable products — Determination of chloride content

1 Scope and field of application

This International Standard specifies a method for the determination of the chloride content of vegetable products.

1.1 Special case

If the product contains natural anthocyanin pigments, the method is applicable with the modifications specified in clause 8.

2 Definition

chloride content of vegetable products : The total chloride content determined according to the method specified and expressed as a percentage by mass of sodium chloride.

3 Principle

Precipitation of chloride by the addition of an excess of a standard volumetric silver nitrate solution and titration of this excess of silver nitrate with a standard volumetric potassium thiocyanate solution.

4 Reagents

All reagents shall be of recognized analytical grade. The water used shall be distilled water or water of at least equivalent purity.

4.1 Nitrobenzene.

4.2 Nitric acid, about 4 N solution.

Mix one volume of nitric acid (ϱ_{20} 1,39 to 1,42 g/ml) with three volumes of water.

4.3 Silver nitrate, 0,100 0 N standard volumetric solution.

Dry silver nitrate ($AgNO_3$) for 2 h at 150 °C and allow to cool in a desiccator. Dissolve 16,989 0 g of the dried silver nitrate in water and dilute to 1 000 ml in a volumetric flask.

4.4 Potassium thiocyanate, 0,1 N standard volumetric solution.

Dissolve 9,72 g of potassium thiocyanate (KSCN) in water and dilute to 1 000 ml in a volumetric flask.

Standardize this solution with the silver nitrate solution (4.3) in the presence of the ammonium iron(III) sulphate solution (4.5).

4.5 Ammonium iron(III) sulphate, $[(NH_4)_2SO_4.Fe_2(SO_4)_3.24H_2O]$, saturated aqueous solution, acidified with nitric acid (5 ml of nitric acid, ϱ_{20} 1,39 to 1,42 g/ml, per 100 ml of the solution).

5 Apparatus

Usual laboratory apparatus, and in particular :

5.1 Homogenizer, or **mortar** [for thick, pasty or solid products (6.1.3)].

5.2 Beaker, 250 ml.

5.3 Volumetric flask, 250 ml, complying with ISO 1042.

5.4 Pipettes, one-mark, to deliver respectively 1 — 3 — 5 — 20 ml, complying with ISO 648.

5.5 Conical flask, 200 ml.

5.6 Burettes, 25 ml, complying with ISO/R 385.

6 Procedure

6.1 Preparation of test sample

6.1.1 Products containing distinct solid and liquid phases

If a specification exists, carry out the determination on the phase indicated in the specification. If there is no specification, and in the case of freshly prepared products, mix the laboratory sample thoroughly and carry out the determination on the homogenized sample.

6.1.2 Liquid products

Mix the laboratory sample thoroughly.

6.1.3 Thick, pasty or solid products

Grind the laboratory sample in a homogenizer or mortar (5.1). If necessary, cut the product into small pieces before grinding. Mix the ground sample thoroughly.

6.2 Test portion

Weigh, to the nearest 0,01 g, in the 250 ml beaker (5.2), about 25 g of the test sample (6.1).

6.3 Determination

6.3.1 Preparation of test solution

Add to the test portion (6.2) 100 ml of hot water and mix the contents of the beaker to obtain a uniform consistency. Heat the contents of the beaker to boiling point and boil for 1 min.

Cool, transfer quantitatively into a 250 ml volumetric flask (5.3) and dilute to the mark with water.

Mix thoroughly, allow to stand for 15 min, then filter through a fluted filter paper, collecting the filtrate, F, in a dry vessel.

6.3.2 Titration

Transfer 20 ml of the filtrate F (6.3.1), by means of a pipette (5.4), into the conical flask (5.5) and add 5 ml of the nitric acid (4.2) and 5 ml of the ammonium iron(III) sulphate solution (4.5).

Add, by means of the burette (5.6), a volume (V_1) of the silver nitrate solution (4.3) sufficient for an excess of 5 to 10 ml of the silver nitrate solution to be present after the precipitation of chloride.

Add 3 ml of the nitrobenzene (4.1) and shake the contents of the flask vigorously to coagulate the precipitate.

WARNING — Special precautions are necessary when using nitrobenzene, owing to its toxicity.

Back-titrate the excess of silver nitrate with the potassium thiocyanate solution (4.4) until a brown-pink colour is obtained which persists for 5 min. Record the volume (V_2) of the potassium thiocyanate solution used.

6.3.3 Number of determinations

Carry out two determinations on test portions taken from the same test sample (6.1).

7 Expression of results

7.1 Method of calculation and formula

The chloride content, expressed as a percentage by mass of sodium chloride, is given by the formula

$$0{,}005\,845\,(V_1 - V_2) \times \frac{100}{m} \times \frac{V_3}{V_4}$$

$$= \frac{0{,}584\,5\,(V_1 - V_2) \times V_3}{m \times V_4}$$

where

V_1 is the volume, in millilitres, of the silver nitrate solution (4.3) used (see 6.3.2);

V_2 is the volume, in millilitres, of the potassium thiocyanate solution (4.4) used (see 6.3.2);

V_3 is the volume, in millilitres, to which the filtrate is diluted (see 6.3.1);

V_4 is the volume, in millilitres, of the aliquot portion of the diluted filtrate taken for titration (see 6.3.2);

m is the mass, in grams, of the test portion (6.2).

NOTES

1 If the concentration of the potassium thiocyanate solution is not exactly 0,1 N, a suitable correction factor should be used for V_2 in the calculation of the result.

2 If the procedure specified in clause 6 has been followed exactly, V_3 = 250 ml and V_4 = 20 ml; the formula given above is then reduced to

$$\frac{7{,}306\,25\,(V_1 - V_2)}{m}$$

Take as the result the arithmetic mean of the two determinations, if the conditions of repeatability (see 7.2) have been satisfied.

Express the result to two decimal places.

7.2 Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst on the same test sample shall not exceed 0,05 g of sodium chloride per 100 g of product.

8 Special case : products containing anthocyanin pigments

Anthocyanin pigments present in the filtrate interfere in the titration; it is therefore necessary to eliminate them by decomposition with permanganate. The method shall therefore be supplemented by the following procedure.

8.1 Reagents

In addition to the reagents specified in clause 4 :

8.1.1 Potassium permanganate, saturated solution (about 6,5 g of $KMnO_4$ per 100 ml of water).

8.1.2 Sodium nitrite or **potassium nitrite,** crystalline.

8.2 Procedure

8.2.1 Proceed as in 6.1 to 6.3.1 inclusive.

8.2.2 Transfer 20 ml of the filtrate F (6.3.1), by means of a pipette (5.4), into the conical flask (5.5). Add about 20 ml of the nitric acid (4.2) and, by means of a pipette, exactly 20 ml (V_1) of the silver nitrate solution (4.3).

Heat to boiling point and boil gently for 2 to 3 min.

Add, in 0,5 to 1 ml increments, about 5 to 10 ml of the potassium permanganate solution (8.1.1), while boiling gently.

The liquid should become colourless. If it does not, add a few crystals of sodium nitrite or potassium nitrite (8.1.2) until a colourless liquid is obtained. Continue to boil for 5 min after the solution has become decolorized.

Cool. Add 5 ml of the ammonium iron(III) sulphate solution (4.5).

Continue as described in the fourth paragraph of 6.3.2. (The addition of nitrobenzene is unnecessary.)

9 Test report

The test report shall show the method used and the results obtained. It shall also mention all operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances which may have influenced the results.

The report shall give all details required for the complete identification of the sample.

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